BRITISH STANDARD

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12873-4:2006

Influence of materials on water intended for human consumption — Influence due to migration —

Part 4: Test method for water treatment membranes

The European Standard EN 12873-4:2006 has the status of a British Standard

ICS 13.060.20; 67.250

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 12873-4:2006.

The UK participation in its preparation was entrusted to Technical Committee EH/6, Effects of materials on water quality, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Influence of materials on water intended for human consumption - Influence due to migration - Part 4: Test method for water treatment membranes

Influence des matériaux sur l'eau destinée à la consommation humaine - Influence due à la migration - Partie 4 : Méthode d'essai des membranes des systèmes de traitement d'eau

Einfluss von Materialien auf Wasser für den menschlichen Gebrauch - Einfluss infolge der Migration - Teil 4: Prüfverfahren für Membranen für die Wasserbehandlung

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CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 12873-4:2006) has been prepared by Technical Committee CEN/TC 164 “Water supply”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2006, and conflicting national standards shall be withdrawn at the latest by September 2006.

It is derived from a CEN Workshop Agreement prepared by the Project Team of the CEN Workshop CWA Water Treatment Membranes.

This standard is one of a series of standards on test methods which support the appropriate standards.

It has been drawn up with the objective to describe a test method to determine the migration of substances from water treatment membranes.

Annex A, which is informative, provides a flow diagram of the steps in the test procedure.

Annex B, which is informative, describes a test rig.

This standard is the fourth in a series of standards dealing with the influence of migration from materials on water intended for human consumption, including:

— Part 1 Test method for non-metallic and non-cementitious factory made products;

— Part 2 Test method for non-metallic and non-cementitious site-applied materials;

— Part 3 Test method for ion exchange and adsorbent resins;

— Part 4 Test method for water treatment membranes.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

In respect of potential adverse effects on the quality of water intended for human consumption caused by the materials, it is called to mind that, while awaiting the adoption of verifiable European acceptance criteria, the relevant national regulations remain in force.

1 Scope

This European standard describes a test method for laboratory evaluation of possible adverse effects of water treatment membranes on drinking water quality.

In principle it is applicable to microfiltration, ultrafiltration, nanofiltration, reverse osmosis and electrodialysis modules for use in the treatment of public water supplies and of water inside buildings.

NOTE Such devices can vary considerably in design and operation and hence some modification of the procedures may be required.

Evaluation of the efficiency of the membrane filter in removing contaminants from the treated water is not included.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
analytical sample
portion of the extract collected for the determination of specified water quality parameters

3.2
cell pair
basic unit of ED systems consisting of a cation transfer membrane, a dilute flow (product) spacer, an anion transfer membrane and a concentrate (waste) spacer

3.3
electrodialysis
process in which ionic species in a feed water are transferred by means of an electrical driving force via a membrane, which is either cation or anion specific, to a waste water stream

3.4
feed water
inlet water for the test rig

3.5
leaching test/migration test
determination of the effects on parameters of water quality following contact with the test sample under specified conditions

3.6
leaching/migration
process of transferring constituents from a material of the test sample into test water carried out under specified conditions

3.7
leachate/extract
test water after exposure to the test sample

3.8
membrane module
filtration element including the membrane itself and all parts of its housing and fittings

3.9
procedural blank
test water after contact with the test rig but not with the test sample under the same conditions used for the leaching test

3.10
product
manufactured item, in its finished form, that comes into contact with drinking water

NOTE In case of membrane water treatment systems the product is a membrane module.

3.11
product water
treated water from the water treatment unit

3.12
reject water
tangential water that does not flow through the membrane

3.13
test rig
equipment, which is not part of the test sample, used to perform the test and control test conditions

3.14
test sample
product, or part of a product, submitted for testing for suitability for use in contact with water intended for human consumption

3.15
test water
water used in contact with the test sample for evaluation of migration behaviour

4 Symbols and abbreviations

ED Electrodialysis

5 Test method

5.1 Principle

Water of specified quality is run through a membrane module under specified operating conditions representing as much as possible the actual conditions of use. The procedure comprises several steps including flushing, operation with and without recycling and cleaning and/or disinfection, the latter depending on the manufacturers recommendations. The purpose of the recycling phase is to concentrate the potential contaminants leaching from the test sample. Samples of the concentrates and the single-pass extracts are taken for analysis.

The operating test temperature is maintained at the highest temperature approved by the manufacturer. In the absence of a recommended temperature it is (25 ± 5) °C.

A schematic diagram of the test protocol is shown in Annex A.

Water treatment devices based on membrane technology can be different in design and operation and therefore the procedures described, such as operation conditions and cleaning, may need to be modified in some cases.

5.2 Reagents and apparatus

5.2.1 General

Only reagents of analytical grade shall be used, except where specified otherwise. All reagents shall be of sufficient purity to ensure that they do not give rise to interferences during the analysis of the extracts.

NOTE Contamination can arise from various sources, e.g. plastics or rubber materials. The use of procedural blanks and laboratory blanks assists in detecting any contamination and identifying its source.

5.2.2 Test water

The test water shall consist of purified water conforming to grade 3 of EN ISO 3696 with an electrical conductivity not exceeding (10 ± 0,1) µS/cm. It shall be free from organic contaminants that can interfere with the analysis of the extracts.

If testing with chlorinated test water is required then use test water according to 5.2.2 having an active chlorine content of (1 ± 0,2) mg/l as Cl_2.

When testing electrodialysis modules the test water shall consist of purified water containing (1 ± 0,1) g/l of NaCl.

NOTE Purification of water, such as tapwater, by a sequence consisting of carbon filtration, reverse osmosis or distillation and followed by deionization usually produces satisfactory test water.

5.2.3 Flushing water

The flushing water should be of a similar quality to the test water (5.2.2). Good quality tap water may be used provided that the following conditions are complied with:

— total organic carbon content of (<1,0 mg/l ± 0,1 mg/l) carbon;

— conductivity of <500 µS/cm;

— it is free from organic contaminants that can interfere with the analysis of the extracts;

— flushing is finished with a volume of the test water, corresponding to (5 ± 0,5)% of the total volume used for flushing.

For products not intended for use in contact with chlorinated water only unchlorinated flushing water shall be used.

5.2.4 Cleaning agent

The type and use of agents to clean and/or disinfect the test sample shall be as recommended by the manufacturer.

5.2.5 Procedural blank

Before testing the filtration unit, bypass the test sample or replace it by an inert device (e.g. a piece of pipe made from the same material as the main test rig component, e.g. stainless steel). Fill the test rig with test water and subject the system to a twenty-four hour period of re-circulating flow in accordance with 5.4.3.1.

Collect procedural blank analytical samples (R_0) in accordance with 5.4.3.3.

Run the remaining extract to waste. Flush the test rig by circulating clean test water for (20 ± 5) min and run to waste. Repeat this flushing once more. Fill the test rig with fresh test water. Adjust the flow of the test water through the test rig to the minimum operational rate recommended for the test sample. Collect procedural blank analytical samples (T_0) in accordance with 5.4.3.2.

NOTE In the case where the complete water treatment unit is to be tested and no other parts are added that would be in contact with the test water, this stage may be omitted. Test water itself should then be analyzed as the procedural blanks R_0 and T_0.

5.2.6 Test rig

The test rig used is dependant on the device under test. Therefore the design and operation of the test rig for the membrane module under test shall reproduce as far as practical the conditions of actual use in service. In particular, testing shall be carried out under the same conditions, as for actual use, for the following:

a) start-up procedure;

b) use of cleaning and/or disinfection agents;

c) water treatment membrane operating conditions, as relevant.

As an example for a membrane system the test rig may include a pump, a reservoir of test water, pressure gauges, flow meters, sampling taps and connecting pipes and fittings. The rig shall allow temperature control, sampling of the product water, re-circulation of the product water and, where relevant, of the reject water or concentrate over a period of 24 h. The re-circulation reservoir should hold sufficient volume of the feed water to allow at least 30-minutes operation at the minimum recommended flow. The different parts of the test rig shall be inert towards the test water used.

NOTE A schematic diagram of an example test rig is shown in Annex B.

5.3 Test samples

5.3.1 General requirements

Test samples shall be taken from normal production batches.

Where a test sample cannot be taken from the production batch an alternative sample is permitted together with any necessary changes to the test procedure. Such modifications shall be fully described in the Test Report.

Test samples shall be representative of production batches and shall have received no other treatment, unless part of the commercial procedure.

The supplier's product manual, as supplied to customers, shall be consulted for the procedures for installation, flushing, chemical treatment and/or disinfection and operation. The following information, depending on the particular membrane device, is required:

a) effective surface area of the membrane;

b) dead volume of the membrane module;

c) minimum and typical product water flows and operating pressure;

d) range of operating reject water flows and pressures;

e) details of recommended start-up procedure;

f) details of recommended disinfection and/or cleaning procedure;

g) data showing the removal of the chemicals during the recommended procedures e) and f).

5.3.2 Sample storage

Test samples shall be transported and stored as specified in the supplier's product manual.

NOTE To prevent dehydration and growth of bacteria, filter elements are usually saturated with a solution of a preservative.

5.4 Procedure

NOTE The analysis and number of tests to be carried out, e.g. single tests or duplicates, will be specified in national or other regulations. As guidance, the method assumes single testing. Duplicate tests should be carried out at the same time using two test rigs or sequentially on the same test rig, provided that the second test is preceded by another procedural blank test.

5.4.1 Test temperature

Ensure that the operating temperature is maintained at the highest temperature recommended by the manufacturer. In the absence of a recommended temperature it shall be (25 ± 5) °C.

5.4.2 Start up procedure

After connecting the membrane module to the test rig, run flushing water (5.2.3) through the module to waste at the flow rates, and over a time period, specified by the manufacturer. The procedure may include normal air scrubbing, permeation/filtration and reverse flushing or cross-flow flushing as specified. If disinfection is required at the point of installation carry out the procedure in accordance with the manufacturer's recommendations. On at least three occasions collect analytical samples (FLi) of the product water flow and determine the residual concentration of the presevative(s).

Do not start the leaching test unless the concentration of the residual preservative at the end of the specified flushing period is below the manufacturers specified value.

NOTE This phase of the test is designed to check the efficiency of the flushing procedure recommended by the manufacturer to remove the conditioning chemicals (usually preservatives) before connecting the unit to the water supply. The aim is to control the start of the leaching test such that the condition of the test sample corresponds to that of the product when the outflow would be introduced into the water supply under field conditions.

5.4.3 Leaching test

The following procedure may need to be modified for some types of membrane devices.

5.4.3.1 First recycling period

At the end of the start up sequence, circulate the test water (5.2.2), held in a reservoir, through the test module. Adjust the volume (m^3) of the test water to approximately one half of the product water flow (m^3/h) through the module so that the test water passes 48 times through the unit during the (24 ± 1) h period. For products operating at tangential flows such as reverse osmosis or ED modules, recycle both the product water and the reject water while maintaining the 48 passes through the test sample. The product water should be re-circulated through the feed water reservoir at flows set to the minimum recommended operational rates.

5.4.3.2 Collection of initial single-pass analytical samples (T1)

During the first 30 min of recycling collect a volume of the product water flow (leachate) equivalent to at least 20 l or one volume of the filtration unit, whichever is smaller, into one clean glass or stainless steel vessel. Transfer portions of the leachate (T_1) into sampling bottles in accordance with the analysis to be carried out.

NOTE The samples represent outflow from the filter at the time of its introduction to the water supply.

5.4.3.3 Collection of analytical samples of the concentrated extract (R1)

After (24 ± 1) h, at the end of the first recycling period, collect analytical samples (R_1) of the extract concentrated by re-circulation in the test rig.

NOTE 1 The concentrated leachates could contain high concentrations of compounds leaching from the test sample. Sampling vessels for the single-pass sampling, T, and for the concentrated leachates, R, should, therefore, be kept separately to avoid cross contamination.

NOTE 2 The recycling step allows an increase in the sensitivity of the test and, therefore, enables the detection of lower concentrations of substances released from the filtration unit.

5.4.3.4 Second recycling period

At the end of the first recycling period run the remaining concentrated leachates to waste. Flush the test rig and the module by circulating clean test water for (20 ± 5) min and run to waste. Repeat this flushing once more. Fill the test rig with fresh test water. Repeat the procedure given in 5.4.3.1.

5.4.3.5 Collection of second single-pass analytical samples (T_2)

Repeat the procedure given in 5.4.3.2.

5.4.3.6 Collection of analytical samples of the second concentrated extract (R_2)

At the end of the second recycling period collect analytical samples (R_2) of the extract concentrated by re-circulation in the test rig.

NOTE For products where substances were detected in the R_1 extracts, the second recycling step would provide information on the changes of leaching rates with time.

5.4.3.7 Collection of third single-pass analytical samples (T_3)

At the end of the second recycling period run the remaining concentrated extract to waste. Flush the test rig and the module by circulating test water for (20 ± 5) min and run to waste. Repeat this flushing once more. Fill the test rig with test water. Set the product flow rate to the minimum operational flow rate recommended by the manufacturer. Collect a sample (T_3) of the product water as described in 5.4.3.2.

NOTE The inclusion of the two recycling periods between the three single-pass sampling occasions provides a more realistic representation of operation under service conditions. Analysis of the three extracts T_1 to T_3 provides information on the changes of leaching rates with time.

5.4.3.8 Sample coding

Code the analytical samples collected as listed in Table 1

Table 1 — Analytical samples from the test

Test sample	Stage	Clause	Leachate code
Membrane module (a)	Procedural blank	5.2.5	R_0/a
Test rig without test sample		5.2.5	T_0/a
	Initial flushing of preservative	5.4.2	FL1,2..ia [a]
	Leaching test	5.4.3.2	T_1/a
		5.4.3.3	R_1/a
		5.4.3.5	T_2/a
		5.4.3.6	R_2/a
		5.4.3.7	T_3/a
Membrane module (b)	Procedural blank	5.2.5	R_0/b
		5.2.5	T_0/b
	Initial flushing	5.4.2	FL1,2..i/b[a]
	Leaching test	5.4.3.2	T_1/b
		5.4.3.3	R_1/b
		5.4.3.5	T_2/b
		5.4.3.6	R_2/b
		5.4.3.7	T_3/b
[a] i ≥ 3 (5.4.2)			

5.4.4 Cleaning and disinfection

5.4.4.1 General requirement

The following procedure shall be carried out on test samples where chemical cleaning and/or disinfection are part of the routine operation of the product. The same test sample may be used after the completion of the leaching test (5.4.3). In such a case the cleaning/disinfection stage shall be carried out as soon as the step in 5.4.3 is completed.

The conditions of the disinfection/cleaning step in the test shall be as close as possible to the operating conditions recommended by the manufacturer. The information required includes the nature of the disinfection agent to be used for the product (e.g. chlorine, hydrogen peroxide, peracetic acid), its concentration in the working solution, contact time and temperature during the treatment, and conditions of the flushing regime (i.e. flow and duration). The maximum recommended operational temperature shall be used for the test.

NOTE 1 Normally using only one, the most aggressive, of the recommended agents (usually chlorine or hydrogen peroxide) is sufficient.

Bypass or remove the test sample, replacing it with an inert device (5.2.5) and carry out the cleaning/disinfection procedure, in accordance with the recommended conditions, followed by flushing using flushing water as specified in 5.2.3. Prepare procedural blank samples DR_0 and DT_0 in accordance with 5.2.5.

NOTE 2 In cases where the complete water treatment unit is to be tested and no other parts are added that would be in contact with the test water, this stage may be omitted. Test water itself should then be analyzed as the procedural blanks DR_0 and DT_0.

Replace the inert device with the test sample and carry out the cleaning/disinfection procedure in accordance with the recommended operating conditions. During the flushing period, on at least three occasions, collect analytical samples (DFLi) of the filtrate/permeate and determine the residual concentration of the disinfectant(s). The residual concentration shall not exceed that specified by the manufacturer.

NOTE 3 This phase of the test is designed to evaluate whether the cleaning/disinfection procedure gives rise to new contaminants or increased leaching of the original contaminants from the product.

NOTE 4 For some membrane devices it may be inappropriate (they may deteriorate) to store them during the period when the rig is being cleaned/disinfected. In such cases the cleaning/disinfection step may be carried out immediately after 5.2.3 has be completed.

5.4.4.2 Post-cleaning recycling period

At the end of the post-cleaning flushing, circulate the test water (5.2.2) through the module as described in 5.4.3.1.

5.4.4.3 Collection of post-cleaning single-pass analytical samples (DT_1)

Repeat the procedure given in 5.4.3.2.

5.4.4.4 Collection of analytical samples of the post-cleaning concentrated extract (DR1)

Repeat the procedure given in 5.4.3.3.

5.4.4.5 Sample coding

Code the analytical samples collected as listed in Table 2

Table 2 — Analytical samples from post cleaning leaching check

Test sample	Stage	Clause	Leachate code
Membrane module (a)	Procedural blank	5.4.4.1	DR_0/a
		5.4.4.1	DT_0/a
	Disinfectant flushing	5.4.4.1	DFL1,2..i/a[a]
	Leaching test	5.4.4.3	DT_1/a
		5.4.4.4	DR_1/a
Membrane module (b)	Procedural blank	5.4.4.1	DR_0/b
		5.4.4.1	DT_0/b
	Disinfectant flushing	5.4.4.1	DFL1,2..i/b[a]
	Leaching test	5.4.4.3	DT_1/b
		5.4.4.4	DR_1/b

[a] $i \geq 3$ (5.4.4)

6 Analysis

Analyze the analytical samples and procedural blanks as specified for the product.

NOTE 1 The range of analyzes to be applied to the analytical samples, the required analytical performance and the interpretation of the resulting data will be specified in national or other regulations.

In case of ED modules conductivity shall be also determined in all leachates and procedural blanks in order to check for carry over from the cell pairs.

NOTE 2 Preparation and analysis of positive controls (test water spiked with the specific determinants and kept under the conditions of the test) is only required if the samples concentrated by recycling (R) are to be analyzed for specific determinants.

If extracts are not analyzed immediately, then the storage time and conditions shall be such that they do not adversely affect the analytical result. Carry out the required analysis on the extracts using analytical methods capable of the required selectivity, sensitivity and accuracy for each specified determinant.

NOTE 3 General guidance on the analytical performance requirements such as detection limit and accuracy is contained in ENV ISO 13530.

Evidence of the performance of the analytical methods used, shall be included in the test report (Clause 8). Evidence shall be provided for the following:

— detection of the determinant present at the lowest concentration of interest;

— an adequately high recovery rate (where appropriate) which is consistent over the concentration range of interest;

— the analytical method does not suffer from serious interferences;

— the determinant is stable under the conditions and over the intended period of storage between sampling and analysis.

7 Expression of results

7.1 Concentrations in the extracts

Concentrations of specific determinants shall be corrected for recoveries and blanks, as appropriate. Any calculations and the values used to arrive at the reported concentrations shall be included in the test report.

7.2 Daily migration rates

7.2.1 Calculation based on concentrations in extracts from one-pass sampling, T_1 to T_3

Calculate the daily migration rate, M_{Ti}, in micrograms per decimetre squared per day [µg/(dm$^2\times$ day^{-1})] from equation 1:

$$M_{Ti} = 0{,}24 \times \left(C_{Ti} \times F_{Ti}\right) \qquad (1)$$

where

C_{Ti} is the concentration of the determinant in a single-pass leachate Ti in micrograms per litre (µg/l);

F_{Ti} is the actual product water flow rate through the membrane module at the time of sampling leachate Ti in litres per square metre per hour [l/[m$^2\times$h^{-1})].

i denotes the migration period T (T_1 to T_3)

7.2.2 Calculation based on concentrations in leachates after re-circulation, R_1 and R_2

Calculate the daily migration rate, M_{Rj} , in micrograms per decimetre squared per day [µg/(dm^2 ×day^{-1})], from equation 2:

$$M_{Rj} = \frac{\left(24 \times C_{Rj} \times V_{Rj}\right)}{\left(S \times T_{Rj}\right)} \qquad (2)$$

where

C_{Rj} is the concentration of the determinant in the concentrated leachate, Rj in micrograms per litre (µg/l);

V_{Rj} is the volume of the re-circulating test water in stage R_j in litres (l);

S is surface area of the membrane in the test sample in decimetres squared (dm^2);

T_{Rj} is the duration of the re-circulation period R_j in hours (h)

j denotes the leaching period R (R_1 and R_2)

8 Test report

The test report shall include the following information:

a) reference to this document;

b) name and the address of the laboratory performing the testing;

c) date of the test sample reception;

d) unique laboratory number;

e) date of the start of the test;

f) trade name and reference of the product;

g) name of the manufacturer of the product, the production place and date;

h) proposed use of the product;

i) details of the test rig, including a schematic diagram;

j) conditions of the initial flushing period including its duration, flow rates and total volume of water passed through the membrane module, and records of the monitoring of removal of the preservative;

k) conditions of the recycling periods;

l) conditions of the cleaning/disinfection period, including details of the chemicals used and conditions of the flushing period as in j);

m) test temperature;

n) methods of analysis used and the source of the methods, including the detection limit and estimates of accuracy;

o) estimated concentrations in the extracts, presented in tabular form, and any calculations used, such as blank and/or recovery corrections;

p) calculated daily migration rates presented in tabular form.

Annex A
(informative)

Diagram of the test procedure

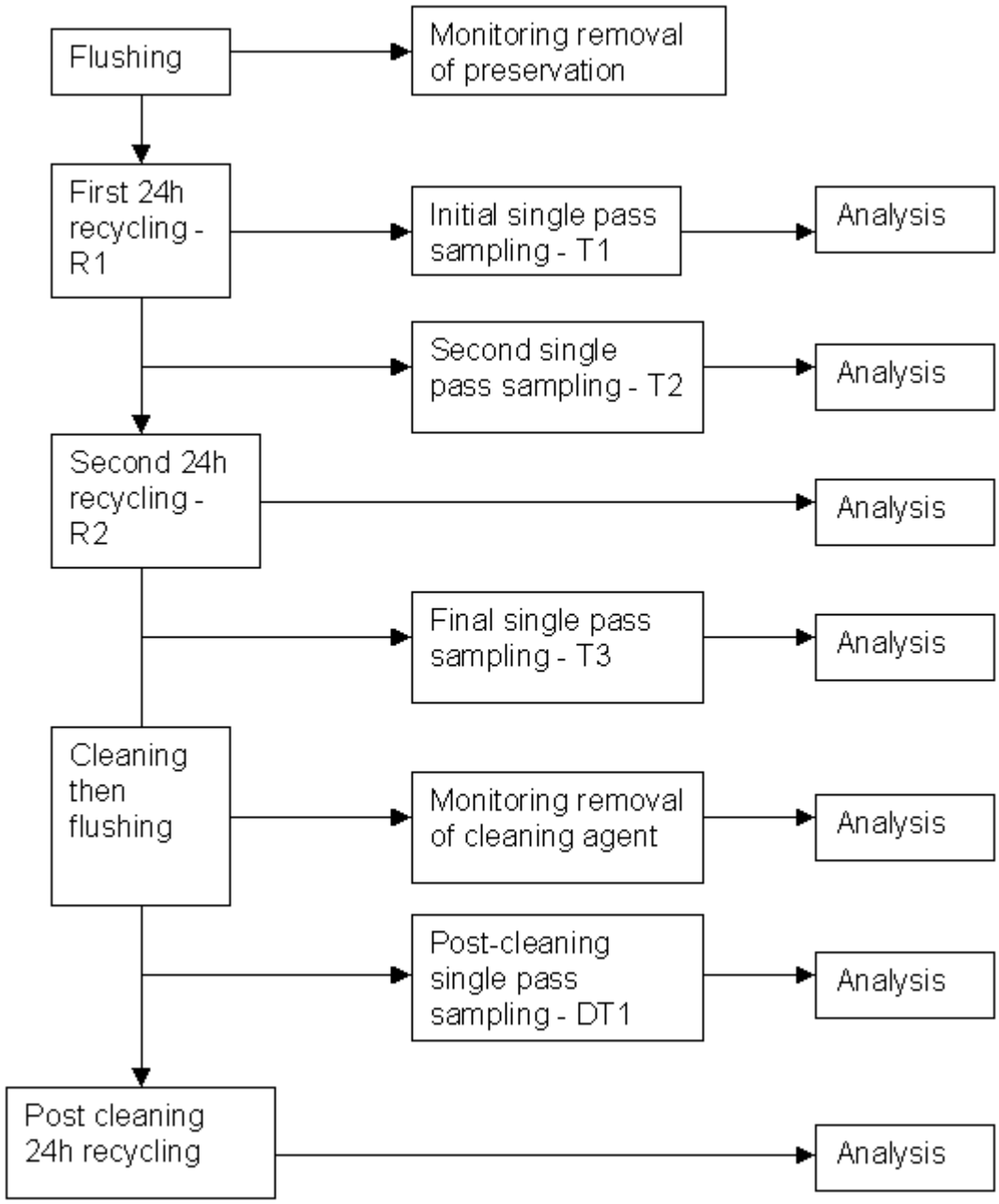
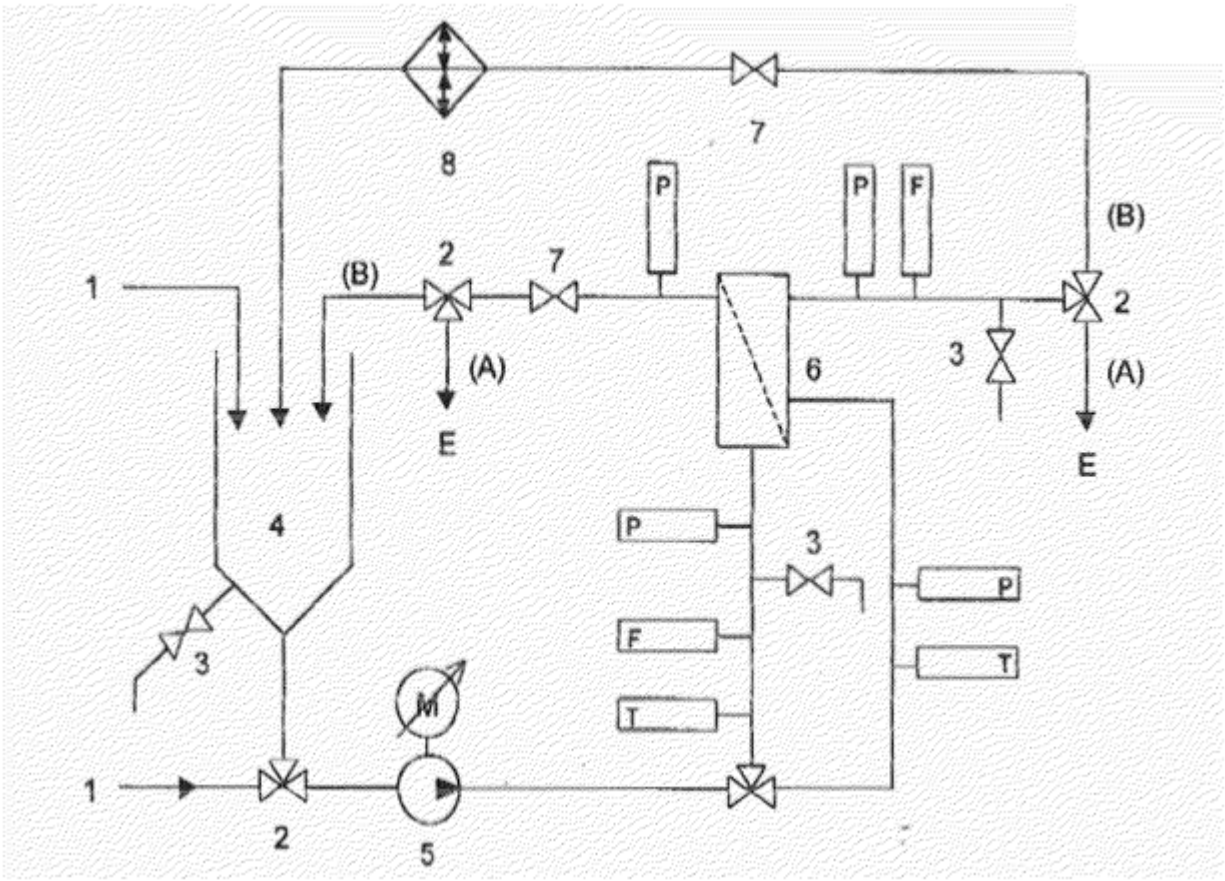


Figure A.1

Annex B
(informative)

Schematic diagram of an example test rig



Key

1	Test water	(A)	One pass circuit
2	Three ways ball valve	(B)	Recycling circuit
3	Sampling tap	E	Drain
4	Test reservoir	F	Flow meter
5	Pump	P	Pressure gauge
6	Test membrane module	T	Temperature gauge
7	Counter pressure regulation valve		
8	Heat exchanger		

Figure B.1

Bibliography

[1] ENV ISO 13530:1998, *Water quality - Guide to analytical quality control for water analysis (ISO/TR 13530:1997)*

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